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# Environmental Science

*An Indian Journal*


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*Current Research Paper*

ESAIJ, 2(3), 2007 [211-217]

## Solid Phase Extraction Of Trace Amounts Cu, Fe And Pb As Bis(2-Hydroxyphenylamino) Glyoxime Complexes On Naphthalene Adsorbent And Flame Atomic Absorption Determination

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Received: 29<sup>th</sup> April, 2007 ; Accepted: 4<sup>th</sup> May, 2007

### ABSTRACT

A simultaneous preconcentration method was developed for determination of trace amounts of Cu, Fe and Pb by atomic absorption spectrometry. The method is based on the retention of their bis(2-hydroxyphenylamino)glyoxime(oxime I) complexes by naphthalene methyltrioctyl ammonium chloride adsorbent in a column. The adsorbed metal complexes were eluted from the column with nitric acid and Cu, Fe and Pb were determined by flame atomic absorption spectrometry. Several parameters such as pH of the sample solution, ligand concentration, volume of the sample and the amount of methyltrioctyl ammonium chloride loaded on naphthalene were evaluated. The effect of diverse ions on the preconcentration was also investigated. A preconcentration factor of up to 100 or more can easily be achieved depending on the volume of the sample taken. The calibration graphs were obtained in the range of 5-40, 10-100 and 10-200ng ml<sup>-1</sup> for Cu, Fe and Pb in the initial solution, respectively, when using 500ml of the solution. The detection limit based on three standard deviations of the blank was 2.2, 3.1, and 4.2ng ml<sup>-1</sup> for Cu, Fe and Pb, respectively. The relative standard deviations (R.S.D.) of 1.1-2.7% for Cu, 2.2-3.6% for Fe and 12-2.9% for Pb were obtained. The method was applied to the determination of Cu, Fe and Pb in river and wastewater samples.

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### KEYWORDS

Solid-phase;  
Preconcentration;  
Lead; Iron;  
Copper;  
Bis(2-hydroxyphenylamino)  
glyoxime (oxime I) and  
naphthalene-methyltrioctyl  
ammonium chloride.

### INTRODUCTION

The determination of metal ions in natural samples such as waters, soils and biological fluids is very important part of environmental and public

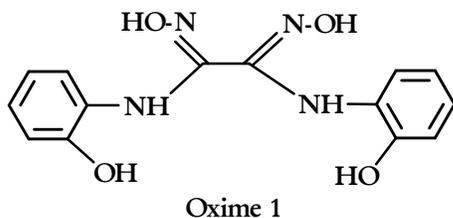
health studies. However, the direct determination of metal ions at trace level is limited due to their low level of concentration and matrix interferences. Flame atomic absorption spectrometry (FAAS) which has been continuously used for the determination of trace

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metal ions suffers from insufficient sensitivity for direct determination of metal ions in environmental samples. Therefore a preconcentration or separation step is frequently necessary to improve the detection limit and sensitivity. For this purpose several separation and preconcentration procedures have been developed for trace metal ion determination involving different analytical strategies. These methods include ion exchange<sup>[1]</sup> liquid extraction<sup>[2]</sup>, anodic stripping voltammetry<sup>[3]</sup>, cloud point<sup>[4]</sup>, coprecipitation<sup>[5]</sup> and solid-phase extractions<sup>[6-10,24-30]</sup>. Among the various methods, solid-phase extraction has received more acceptances due to its simplicity, rapidity and attainability of large preconcentration factor.

A variety of solid materials such as modified ion exchange resins<sup>[11-13]</sup>, functionalized resins with chelating reagents<sup>[14]</sup>, activated carbon<sup>[15]</sup>, zeolites<sup>[16]</sup>, cellulose<sup>[17]</sup> and immobilized microorganisms on sepiolite<sup>[18]</sup> have been used for preconcentration of trace metals. Microcrystalline naphthalene<sup>[19-21]</sup> and benzophenone<sup>[22]</sup> have also been used as solid-phase for adsorptive extraction of metal ion complexes.

In this work a simultaneous solid-phase preconcentration method for the determination of copper, iron and lead by atomic absorption is described. The method is based on the adsorption of their bis(2-hydroxyphenylamino)glyoxime(oximeI) complexes on methyltriocetyl ammonium chloride supported on naphthalene used as an adsorbent in a column. The complexes adsorbed on the naphthalene adsorbent are then eluted with nitric acid solution and Cu, Fe and Pb are determined by FAAS.



## EXPERIMENTAL

### Apparatus

A Philips PU9100X flame atomic absorption spectrometer was used for the determination of copper, iron and lead. It was equipped with appropriate

hallow cathode lamp and air-acetylene burner. The instrumental parameters were as follows: wavelength 324.8, 248.2 and 217.0nm for Cu, Fe and Pb, respectively. The lamp currents of 3, 13 and 7.5mA were used for Cu, Fe and Pb, respectively. Band pass was 0.5nm for three elements. All pH measurements were made using a Metrohm digital pH meter with a combined glass electrode. A Shimadzu rotary oil vacuum pump type SA18 was used.

### Reagents

Doubly distilled de-ionized water and analytical grade reagents were used for the preparation of all the solutions. The stock solution of 1000 $\mu\text{gml}^{-1}$  copper was prepared by dissolving 0.6706 g of  $\text{CuCl}_2 \cdot 5\text{H}_2\text{O}$  (Merck) in water and diluted to 250ml in a volumetric flask. This solution was standardized iodometric procedure<sup>[23]</sup>. The required concentration of copper solution was prepared by appropriate dilution of the stock solution. The stock solution of 1000 $\mu\text{gml}^{-1}$  iron was prepared by dissolving 0.8634 g of  $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$  (Merck) in 100 ml of distilled water containing 10 ml of concentrated hydrochloric acid. This solution was standardized using dichromate procedure<sup>[23]</sup>. More dilute solutions were prepared by appropriate dilution of the stock solution. The stock solution of 1000 $\mu\text{gml}^{-1}$  lead was prepared by dissolving 0.1599g of  $\text{Pb}(\text{NO}_3)_2$  (Merck) in water and diluted to 100ml in a volumetric flask. The required concentration of lead solution was prepared by appropriate dilution of the stock solution. A 3.0mol l<sup>-1</sup> nitric acid solution was prepared by diluting 20ml of  $\text{HNO}_3$  (Merck) to 100ml in a volumetric flask.

Oxime I was synthesized and purified as follows: 40ml of ortho-aminophenol was dissolved in a mixture of 40ml methanol and 67ml ethanol, and 27ml of double distilled water was added. Then, 20mmol dichloroglyoxime dissolved in 20ml methanol was added instantly, and the obtained solution was stirred. A white brown precipitated product will have appeared slowly after 5min. After addition of 20mmol sodium bicarbonate, the mixture was stirred for 3h. Finally, the precipitated product, was filtered out, washed with cold ethanol, and recrystallized from water/methanol(3:1) mixture. The structure of the

compound was confirmed by NMR and FT-IR spectrometry<sup>[31]</sup>.

### General procedure

A funnel-tipped glass tube (80mm length and 8mm i.d.) with a very fine bore was used as a preconcentration column. It was filled with the adsorbent slurry to a height of 7cm after slightly pressing the adsorbent in the column with a flat glass rod. Solution (500ml) containing 5-50, 10-50 and 10-150ng ml<sup>-1</sup> of Cu, Fe and Pb, respectively,  $1.93 \times 10^{-5}$  mol l<sup>-1</sup> bis(2-hydroxyphenylamino) glyoxime (oxime I) and formate buffer solution (pH 4) was passed through the column at a flow rate of 10ml min<sup>-1</sup>. The column packing was then washed with a small volume of water and the naphthalene material was pushed down with a flat glass rod to eliminate the excess water attached to naphthalene. The metal ion complexes were eluted with 5ml of HNO<sub>3</sub> solution (3.0mol l<sup>-1</sup>) and Cu, Fe and Pb were determined by FAAS. A blank solution was also run under the same analytical conditions without adding any Cu, Fe and Pb.

## RESULTS AND DISCUSSION

The preliminary experiments showed that Cu, Fe and Pb complexes with bis(2-hydroxyphenylamino) glyoxime (oxime I), could be simultaneously retained on the methyltrioctyl ammonium chloride-naphthalene adsorbent. Therefore, the retention of these complexes on a column containing methyltrioctyl ammonium chloride-naphthalene adsorbent was chosen for simultaneous preconcentration of Cu, Fe and Pb with subsequent determination by FAAS.

### Effect of pH

The effect of pH of the test solution on the retention of Cu, Fe and Pb was studied. The pH values of solutions containing Cu, Fe and Pb complexes with bis(2-hydroxyphenylamino) glyoxime (oxime I) was adjusted in the range of 2-6 using appropriate buffer solutions and the general procedure was followed. The results of this study showed that maximum retention of the Cu, Fe and Pb was obtained at pH 4 (Figure 1). Thus a formate buffer with pH 5 was selected for further work.

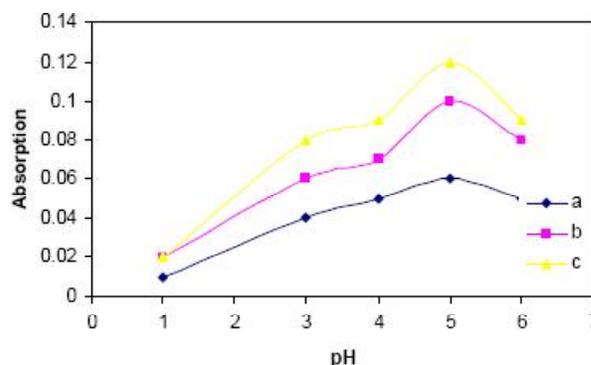


Figure 1: The effect of pH on the absorbance of the (a) 50 ng ml<sup>-1</sup> of Cu, (b) 50 ng ml<sup>-1</sup> of Fe and (c) 150 ng ml<sup>-1</sup> of Pb. Conditions: volume of the sample 100ml, bis(2-hydroxyphenylamino) glyoxime (oxime I) (OXIME1) concentration  $1.93 \times 10^{-5}$  mol l<sup>-1</sup>.

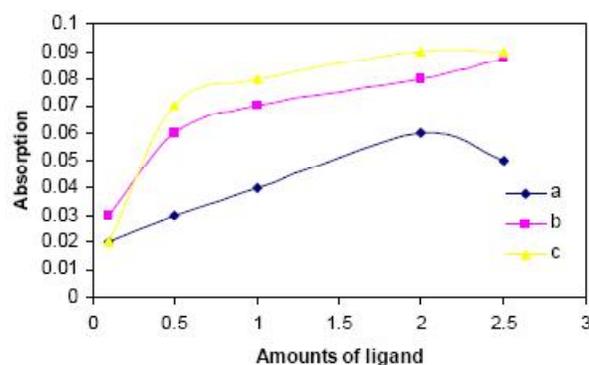


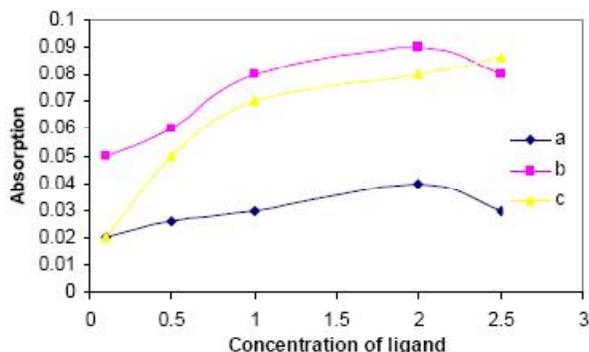
Figure 2: The influence of bis(2-hydroxyphenylamino) glyoxime (oxime I) concentration on the absorbance of (a) 50 ng ml<sup>-1</sup> of Cu, (b) 50 ng ml<sup>-1</sup> of Fe and (c) 150 ng ml<sup>-1</sup> of Pb

Conditions: volume of the sample 100ml, pH 4.

### Effect of oxime (1) concentration

The effect of oxime1 concentration on the retention of the Cu, Fe and Pb complexes was also investigated. The concentration of OXIME1 was varied in a number of 100 ml volumetric flasks containing 50, 50 and 150ng ml<sup>-1</sup> of Cu, Fe and Pb, respectively, and the general procedure was followed. The results shown in figure 2 indicate that the concentration of OXIME1 did not affect the retention of the all copper and iron ions very much. However, the retention of lead was increased with increasing OXIME1 concentration up to  $1.93 \times 10^{-5}$  mol l<sup>-1</sup> and remained constant at higher concentrations. This behavior could probably be due to the difference in the formation constants of the metal ion complexes and the number of moles of the ligand required for each metal ion to form a complex. Therefore, OXIME1 concentration of  $1.93 \times 10^{-5}$  mol

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**Figure 3 :** The effect of the amount of methyltriethyl ammonium bromide loaded on the column: (a) 50ng ml<sup>-1</sup> of Cu, (b) 50ng ml<sup>-1</sup> of Fe and (c) 150ng ml<sup>-1</sup> of Pb. Conditions: Volume of the sample 100ml, pH4, bis(2-hydroxyphenyl amino) glyoxime (oxime I) concentration 1.95×10<sup>-2</sup> mol l<sup>-1</sup>.

l<sup>-1</sup> was selected as optimum.

### Effect of nitric acid as the eluent

Studies were carried out to investigate the influence of different acids as eluents for the retained complexes. Nitric acid was chosen as an eluent owing to its effective elution of the adsorbed complexes. The effect of eluent concentration on the absorbance of eluted solution containing Cu, Fe and Pb was examined. The absorbance of all three ions was increased, as the HNO<sub>3</sub> concentration increased up to 3.0mol l<sup>-1</sup> and it remained constant above this concentration. Therefore a nitric acid concentration of 3.0mol l<sup>-1</sup> was selected for subsequent studies. In order to choose proper volume of the eluent, the retained complexes were stripped with different volumes (3-10ml) of 3.0mol l<sup>-1</sup> nitric acid. 10ml would not be suitable because it gave a smaller preconcentration factor and 3ml was not sufficient for the elution. Hence, 5ml of 3.0mol l<sup>-1</sup> nitric acid was chosen for subsequent elution of the metal ion complexes for more convenient.

### The effect of amount of methyltriethyl ammonium chloride

The amount of methyltriethyl ammonium chloride loaded on naphthalene was optimized and the results are shown in figure 3. A loading of 1g of methyltriethyl ammonium chloride on 10g of naphthalene gave highest absorbance for the three ions.

### Effect of sample and elution flow rate

The retention of Cu, Fe and Pb complexes with OXIME1 on naphthalene adsorbent was not affected by sample flow rates in the range of 2-8 ml min<sup>-1</sup>. Thus the experiments were performed at a sample flow rate of 8mlmin<sup>-1</sup> which was the maximum flow rate attainable by the pump we used. It is the same for the elution flow rate and flow rates in the range of 2-8 ml min<sup>-1</sup> can be applied without any loss in recovery.

### Effect of sample volume

The volume of the aqueous-phase was varied from 25 to 500ml all containing same amounts of Cu, Fe and Pb so that the final solutions after performing the preconcentration procedure would have the same concentrations. It was found that the absorbance was constant in all solutions after following the preconcentration procedure. However, at higher volumes the analysis time is increased. Therefore a preconcentration factor of 100 can be easily achieved when using 500 ml nitric acid concentration of 3.0mol l<sup>-1</sup> was selected for subsequent studies. In order to choose proper volume of the eluent, the retained complexes were stripped with different volumes(3-10ml) of 3.0mol l<sup>-1</sup> nitric acid. 10ml would not be suitable because it gave a smaller preconcentration factor and 3ml was not sufficient for the elution. Hence, 5ml of 3.0mol l<sup>-1</sup> nitric acid was chosen for subsequent elution of the metal ion complexes for more convenient.

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### The sorption capacity

The sorption capacity of the adsorbent was determined by batch process. The adsorbent slurry was filtered on a filter paper and air-dried. Adsorbent(1 g) was equilibrated with 100ml of a solution containing 1, 1 and 5 $\mu$ gml<sup>-1</sup> of copper, iron and lead, respectively, for 24h at optimum conditions. The loading capacity for each metal ion was calculated from the difference between the metal ion concentration before and after adsorption. The values were 100, 100 and 300 $\mu$ g g<sup>-1</sup> of adsorbent for copper, iron and lead, respectively.

### Kinetics of the metal sorption

The rate of loading of Cu, Fe and Pb ions on the adsorbent was determined by batch experiment. Adsorbent(1g) was stirred with 100ml solutions containing 50, 50 and 250ng ml<sup>-1</sup> of copper, iron and lead, respectively, for different time intervals. After a predetermined time, the amount of metal ions loaded was determined using the general procedure. The variation of sorption as a function of time for the metal ions is shown in figure 4. The loading half time  $t_{1/2}$  defined as the time to reach 50% of total loading capacity as estimated from figure 4 is about 7 min for copper and iron and 15 min for lead. It is also observed that an equilibrium time of 15min was required for 95-99% sorption of copper and iron and 30 min for 90% sorption of lead.

### Analytical performance

The calibration graphs were obtained using the recommended procedure under the optimum conditions with a sample volume of 500 ml. Linear graphs were obtained in the range of 5-40, 10-100 and 10-200ng ml<sup>-1</sup> for Cu, Fe and Pb in the initial solution, respectively. (The concentration range in the final solution after preconcentration is in the range of 0.5-5.0, 1.0-5.0 and 1.0-15.0  $\mu$ gml<sup>-1</sup> for Cu, Fe and Pb, respectively.) The equations for the lines were  $A=7.2\times 10^{-3}C-3.6\times 10^{-4}$ , with  $r=0.9995$  for copper,  $A=2.9\times 10^{-3}C+3.0\times 10^{-4}$  with  $r=0.9998$  for iron and  $A=1.1\times 10^{-3}C+1.7\times 10^{-4}$  with  $r=0.9991$ . The detection limit based on three standard deviations of the blank was 2.2, 3.1 and 4.2ng ml<sup>-1</sup> for Cu, Fe and Pb, respectively, when using sample volume of 500ml.

The relative standard deviations(R.S.D.) for 10 replicate measurements of 10 and 30ng ml<sup>-1</sup> of Cu was 2.7 and 1.1%, for 10 and 30ng ml<sup>-1</sup> of Fe was 3.6and 2.0% and for 50 and 150ng ml<sup>-1</sup> Pb was 2.9 and 1.1%.

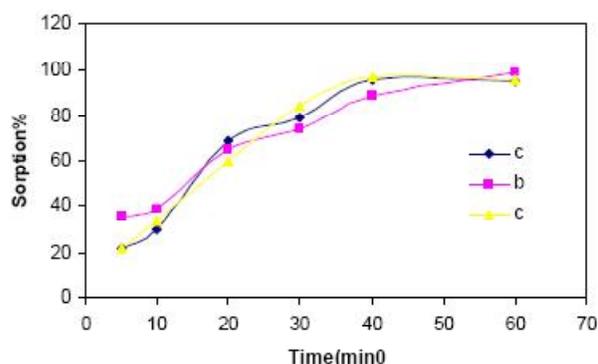
### Effect of foreign ions

The interference of co-existing ions on the determination of Cu, Fe and Pb was studied.

**TABLE 1 : The effect of interfering ions on the recovery of 50, 50 and 150ng ml<sup>-1</sup> of Cu, Fe and Pb**

| Recovery <sup>a</sup> (%) |             |             | Concentration of interfering (mg l <sup>-1</sup> ) | Interfering ion  |
|---------------------------|-------------|-------------|--|--|
| Pb                        | Cu          | Fe          |  |  |
| 101 $\pm$ 3               | 100 $\pm$ 2 | 99 $\pm$ 1  | 1000   | Cd <sup>2+</sup>   |
| 98 $\pm$ 2                | 100 $\pm$ 3 | 100 $\pm$ 2 | 100  | NH <sub>4</sub> <sup>+</sup> , K <sup>+</sup> , Mg <sup>2+</sup> , Li <sup>+</sup> , Cl <sup>-</sup> , NO <sub>2</sub> <sup>-</sup> , SCN <sup>-</sup> , SO <sub>4</sub> <sup>2-</sup> |
| 99 $\pm$ 1                | 99 $\pm$ 2  | 100 $\pm$ 2 | 10   | Hg <sup>2+</sup> , Zn <sup>2+</sup> , Cr <sup>3+</sup> , Ca <sup>2+</sup> , Se(IV), F <sup>-</sup> , Al <sup>3+</sup>  |

<sup>a</sup> $\pm$  ts/v n at 95% confidence (n=3); <sup>b</sup>Masked by NaB<sub>4</sub>



**Figure 4 : Kinetics of metal ions sorption on the adsorbent: (a) 50ng ml<sup>-1</sup> of Fe, (b) 50ng ml<sup>-1</sup> of Cu and (c) 250ng ml<sup>-1</sup> of Pb.**

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TABLE 2 : Determination of Cu, Fe and Pb in water samples

| Lead concentration (ng ml <sup>-1</sup> ) |                    |       | Iron concentration(ng ml <sup>-1</sup> ) |                    |       | Copper concentration(ng ml <sup>-1</sup> ) |                    |       | Sample                                  |
|---|--------------------|-------|--|--------------------|-------|--|--------------------|-------|---|
| Recovery                                  | Found <sup>a</sup> | Added | Recovery                                 | Found <sup>a</sup> | Added | Recovery                                   | Found <sup>a</sup> | Added |   |
| -   | 257 ± 3            | 0     | -  | 68 ± 3             | 0     | -  | 37 ± 1             | 0     | River water<br>(Varamin) <sup>b,c</sup> |
| 99  | 768 ± 9            | 500   | 100                                      | 170 ± 4            | 100   | 100  | 136 ± 3            | 100   |   |
| 100                                       | 1263 ± 16          | 1000  | 100                                      | 270 ± 6            | 200   | 99   | 245 ± 4            | 200   |   |
| -   | 258 ± 3            | 0     | -  | 61 ± 1             | 0     | -  | 29 ± 1             | 0     | seawater <sup>d</sup>                   |
| 99  | 749 ± 9            | 500   | 101                                      | 164 ± 5            | 100   | 96   | 124 ± 3            | 100   |   |
| 101                                       | 1269 ± 16          | 1000  | 100                                      | 256 ± 6            | 200   | 98   | 225 ± 4            | 200   |   |
| -   | 13 ± 1             | 0     | -  | ND                 | 0     | -  | 73 ± 1             | 0     | Tap water<br>(Saveh) <sup>f</sup>       |
| 102                                       | 66 ± 2             | 50    | 100                                      | 10 ± 1             | 10    | 99   | 82 ± 1             | 10    |   |
| 101                                       | 116 ± 2            | 100   | 98                                       | 19 ± 1             | 20    | 99   | 92 ± 1             | 20    |   |

ND(not detected) :  $\bar{x} \pm t_s / \sqrt{n}$  at 95% confidence (n=5) ; <sup>a</sup>A preconcentration factor of 10 was applied ; <sup>c</sup>From a polluted area ; <sup>d</sup>Rolling and Pipe Mills Company ; <sup>f</sup>A preconcentration factor of 100 was applied

Solution(100ml) containing 50, 50 and 150ng ml<sup>-1</sup> of Cu, Fe and Pb, respectively, and various amounts of foreign ions were prepared and the procedure described in the experimental section was applied. Any deviation of ±4% or more from the absorbance value of the standard solution was taken as interference. TABLE 1 shows the results. As can be seen a very good selectivity is achieved.

### APPLICATION

The proposed method was applied to the preconcentration and determination of Cu, Fe and Pb content of tap water (Saveh, 20 February, 2007), seawater(taken from the Caspian sea, Anzali port, 11 November 2006)water samples. The water samples were collected in polyethylene bottles and kept in a refrigerator before use. The seawater sample solution(50 ml) and 500ml for tap water were treated under the recommended procedure. Accuracy of the results was verified by analyzing the spiked water samples. The recoveries for these elements were very satisfactory (TABLE 2) indicating that the method can be used reliably for the analysis of water samples.

### CONCLUSION

Up to our knowledge this is the first time naphthalene adsorbent is used for simultaneous preconcentration of three ions and their determination in the same sample batch. The adsorbent is economical and can easily be prepared. The proposed method for simultaneous preconcentration and sepa-

ration of Cu, Fe and Pb is selective and allows the determination of trace amounts of these elements to be carried out by FAAS which is an available instrument in almost every laboratory. The detection limits achieved are better than some of the previously reported methods using FAAS as detection method<sup>[12,13,18]</sup>. The linear range of the method is sufficient for the determination of Cu, Fe and Pb in some environmental samples and was successfully applied to the determination of copper, iron and lead in tap water and seawater water samples with good recoveries.

### ACKNOWLEDGMENTS

We gratefully acknowledge financial support department of Chemistry, Varamin Campus Islamic Azad University & department of Chemistry Science and Research Campus Islamic AzadUniversity.

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